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IS 12610 (1989): Pesticides - Determination of Residues in Foods - Phosphamidon [FAD 1: Pesticides and Pesticides Residue Analysis]

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Indian Standard

(Reaffirmed 2004)

PESTICIDES — DETERMINATION OF RESIDUES IN FOODS - PHOSPHAMIDON

भारतीय मानक

कीटनाशी — खाद्य पदार्थों में अवशिष्ट ज्ञात करना — फॉस्फामिडॉन

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN. 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 1989

Price Group 1

Pesticides Residue Analysis Sectional Committee, AFDC 56

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 23 February 1989, after the draft finalized by the Pesticides Residue Analysis Sectional Committee had been approved by the Agricultural and Food Products Division Council.

Phosphamidon formulations are extensively used in agriculture for the control of many insect pests. This standard will enable the food, health authorities and others engaged in the field to follow uniform test procedure for the estimation of phosphamidon and its metabolite residues in various foods.

In the preparation of this standard, due consideration has been given to the limits of phosphamidon residues laid under the provisions of ***Prevention of Food Adulteration Rules, 1955*** and the test method is sensitive to the prescribed level of residues.

In reporting the result of a test or analysis made in accordance with this standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

PESTICIDES — DETERMINATION OF RESIDUES IN FOODS — PHOSPHAMIDON

1 SCOPE

1.1 This standard prescribes the gas chromatographic method for the determination of phosphamidon and its major metabolite desethyl phosphamidon residues in foods.

1.2 This method has a detection limit of 0’01 μg/g (0·01 ppm).

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
IS 1070 : 1977	Specification for water for general laboratory use (second *revision)*
IS 11380 : 1985	Method of sampling for the determination of pesticide residues in agricultural and food commodities

3 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (see IS 1070 : 1977) shall be employed in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain -impurities which affect the result of analysis.

4 SAMPLING

The representative samples for the purpose of estimating phosphamidon residues in foods shall be drawn in accordance with IS 11380 : 1985.

5 SAMPLE STORAGE

Samples shall be stored as such when the quantity is small as chances of degradation do not exist. Otherwise, carry out extraction (see 9) and store the extract. Depending upon the nature of the sample, keep the samples or their extracts either in deep freezer at – 15°C or in refrigerator until taken up for analysis. Ensure that the samples do not absorb or lose moisture during storage. Avoid undue long storage periods.

6 METHOD

6.1 Depending on the type of crop, the plant material is macerated and extracted with either acetonitrile or methylene chloride. After addition of water the organic solvent is evaporated, phosphamidon and desethyl phosphamidon are partitioned into chloroform. To remove oily materials from oily crops, the aqueous extract is washed with n-hexane before extraction with chloroform. The chloroform is replaced with n-hexane by repeated additions and evaporations of the latter solvent. The hexane solution is concentrated and applied to a silica gel column which is washed with an ethyl acetare-hexane mixture. Phosphamidon and desethyl phosphamidon are eluted with ethyl acetate. Quantitative determination is done by gas chromatography, using a nitrogen-phosphorus detector (NPD) or a flame photometric detector (FPD) (phosphorus specific). The content of phosphamidon is determined by comparing the response with reference standard of phosphamidon. *Cis* and *trans* phosphamidon and desethyl phosphamidon are eluted simultaneously.

7 APPARATUS

7.1 Mixer-cum-Grinder

7.2 Kuderna Danish Concentrator or Equivalent

7.3 Water-Bath

7.4 Chromatographic Column — 250 mm long, 20 mm ID.

7.5 Soxhlet Apparatus

7.6 Buchner Funnel

7.7 Rotary Vacuum Evaporator

7.8 Gas Chromatograph

Equipped with a NPD or a FPD (phosphorus specific). The following operating parameters are suggested, which may be varied, according to the available facilities, provided standardization is done:

Column	Borosilicate glass, 2 m long and 3·2 mm ID, packed with 3 percent SE-30 on Gas Chrom Q (80-100 mesh)
Temperature	
Column oven	170°C
Injection port	200°C
Detector	170°C
Carrier gas	*Flow Rate*
Nitrogen	40 ml/minute

7.9 Microlitre Syringe

7.10 Waring Blender or Equivalent

8 REAGENTS

8.1 Methylene Chloride, glass re-distilled

8.2 Chloroform, glass re-distilled

8.3 *n*-Hexane, glass re-distilled

8.4 Ethyl Acetate, glass re-distilled

8.5 Silica Gel, column chromatography grade, activated at 110°C for 6 hours

8.6 **Sadium Sulphate**, anhydrous

8.7 **Acetonitrile,** glass re-distilled

8.8 **Benzene**, glass re-distilled

8.9 Phosphamidon Reference Standard, of -known purity

9 EXTRACTION

9.1 Fruits and Vegetables

Place **50 g** of shredded vegetables or fruits in the waring blender and macerate with 100 ml of acetonitrile at high speed for three minutes. Filter the resulting suspension through a 6-cm Buchner funnel (layered with Whatman No. 1 or equivalent filter-paper) by using a slight vacuum. Rinse the jar and filter cake with two 25-ml portions of acetonitrile. Combine the two filterdtes in a round-bottom flask, add 40 ml of distilled water and evaporate the acetonitrile using a rotary evaporator at 50°C. Transfer the aqueous residue into a 100-ml separatory funnel and extract with three 50-ml portions of chloroform; previously used for rinsing the evaporation flask. Collect the three chloroform fractions in a 500-ml Erlenmeyer flask and remove traces of water by adding 5 g of anhydrous sodium sulphate. Remove the sodium sulphate by filteration through a folded filter paper. Rinse the filter paper with 20-ml of chloroform.

9.1.1 Transfer the chloroform extract to a Kuderna Danish evaporator and concentrate to about 5 ml. Remove the chloroform by repeated additions of 10 ml of n-hexane (3 times) and evaporate to 3 ml.

9.2 **Oily Crops**

For residue analysis of oily crops, such as cotton seeds, place 50 g of coarsely-milled material into a thimble of soxhlet apparatus. Extract with 350 ml of methylene chloride for four hours using a 500-ml round-bottom flask. Add 50 ml of distilled water to the extract and remove the methylene chloride by evaporation on a rotary evaporatory at 40°C. Transfer the aqueous residue into a 500-ml separating funnel, add 150-ml of distilled water previously used for rinsing the evaporation flask and extract with two 100-ml portions of *n*-hexane. If emulsion develops, add 2 ml of saturated sodium chloride solution.

9.2.1 Drain the aqueous phase into another 500-ml separatory funnel, extract with three 100-ml portions of chloroform, and collect the chloroform phases in a 500-ml Erlenmeyer flask. Remove traces of water with anhydrous sodium sulphate, concentrate and transfer the dissolved residue in *n*-hexane as given in **9.1.1.**

10 CLEAN UP

Fill the chromatographic column with n-hexane and adjust the flow to 1-2 ml per minute and place 2 g of the silica gel into the column. When the solvent head reaches the top of the silica gel, close the stopcock and carefully add with a pipette the hexane concentrate on top of the column. Rinse the tube of the Kuderna Danish evaporator with two 3-ml portions of 50 percent ethyl acetate in n-hexane, add the washings to the column with an additional (25 ml) of the same organic solvent mixture. Elute phosphamidon residues with 50-ml of ethyl acetate into 100-ml pear-shaped flask using the same flow rate. Reduce the volume of the eluate to 5 ml using the rotary evaporator. Transfer the concentrate to a 10-ml volumetric flask, wash the evaporation flask with small portions of n-hexane and make up the volume. One ml of this concentrate corresponds to 5 g of the sample.

11 PROCEDURE

Inject suitable aliquot (2μl) into the column using a microlitre syringe. Mentify the peak by its retention time and measure the peak area. For reference standard, prepare n-hexane solutions containing one to ten μg/g of phosphamidon.'

12 CALCULATION

Phosphamidon residue,

$$\mu g/g\ (\text{ppm}) = \frac{A_1 \times V_2 \times V_3 \times C}{A_2 \times V_1 \times M} \times f$$

where

A_1 = peak area of the sample;

A_2 = peak area of the phosphamidon standard;

V_1 = volume, in μl, of sample injected;

V_2 = volume, in μl, of standard phosphamidon injected;

V_3 = total volume, in ml, of sample solution;

C = concentration in μg/g of standard phosphamidon solution;

f = recovery factor

$= \frac{100}{\text{Percent mean recovery}}$; and

M = mass, in g, of sample taken for analysis.

NOTE -Percent mean recovery is determined by taking untreated control sample to which known account Qf phosphamidon is added and analysed as described above.

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75
Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices:	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	331 01 31 331 1375
Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola CALCUTTA 700054	36 24 99
Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036	2 1843 3 16 41
Southern : C. 1. T. Campus, IV Cross Road, MADRAS 600113	41 24 42 41 25 19 41 29 16
Western : Manakalaya, E9 MIDC, Marol, Andheri (East) BOMBAY 400093	6 32 92 95

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